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(54) Solvent extraction process

(57) A process for extracting a compound or composition of matter from a raw material containing that compound or composition as a constituent part is described. The process comprises the steps of (1) contacting a sample of the raw material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the raw material.

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SOLVENT EXTRACTION PROCESS

The present invention relates to a solvent extraction process in which a raw material containing a particular compound or composition is treated with an extraction solvent so as to remove at least a proportion of that compound or composition from the raw material.

Processes for extracting a desired compound or composition from a raw or bulk material which contains that compound or composition as a constituent part using a suitable extraction solvent are known in the art. In these known processes, the raw material is contacted with the extraction solvent, often under vigorous mixing conditions so as to facilitate the dissolution of the desired compound or composition into the extraction solvent, and the resulting solvent liquor containing the desired compound or composition is then separated from the raw material for subsequent processing, e.g. distillation to remove the extraction solvent. Multiple extractions may suitably be carried out on the same raw material sample so as to maximise the amount of the desired compound or composition which is extracted from that sample. Typical examples of extraction solvents which have been used in the prior art extraction processes include hexane, methyl acetate, ethyl acetate, acetone and methanol.

Although solvent extraction processes are used on a commercial scale, the extraction solvents which are currently used in these processes are not wholly satisfactory. Thus, when solvents such as hexane are used to extract flavoured or aromatic oils, such as are used in the food and cosmetic industries, from plant matter containing those oils, unwanted materials contained in the plant, e.g. high molecular weight waxes, tend to be eluted along with the desired oil. This problem necessitates subjecting the resultant hexane liquor to further processing in which the unwanted waxes are removed by extraction, e.g. using ethanol. Furthermore, the extraction solvents which are currently in use have fairly high boiling points, and the elevated temperatures which are employed in the distillation process to remove these high boiling solvents from

the extracted material can cause problems. For example, the flavoured or aromatic oils contained in certain plants are complex substances containing a large number of individual compounds some of which are relatively volatile or relatively thermally unstable. Consequently, high distillation temperatures can tend to result in a loss of product either through co-evaporation of the more volatile compounds with the extraction solvent or thermal degradation of the more thermally unstable compounds.

The present invention provides a new solvent extraction process which can be used to extract a wide variety of compounds or compositions from raw or bulk materials of which they form a constituent part. In one particular embodiment, the present invention provides a solvent extraction process which is capable of extracting the flavoured or aromatic oils contained in certain plant materials without eluting the high molecular waxes they contain.

According to the present invention there is provided a process for extracting a compound or composition of matter from a raw material containing that compound or composition as a constituent part, which process comprises the steps of (1) contacting a sample of the raw material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the raw material.

In one particular embodiment, the extraction process of the present invention can be used to extract a natural product, such as a flavoured or aromatic oil, from a plant material containing that product.

Accordingly, the present invention provides a process for extracting a natural product from a plant material containing that product as a constituent part, which process comprises the steps of (1) contacting a sample of the plant material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the plant material.

When used in this specification, the expression "plant material" not only includes materials which are essentially unprocessed and as such are clearly recognisable as being of plant origin, for example bark, leaves, flowers and seeds, but also materials, which although originating from plants, have been subjected to various processes and as such have a form which is somewhat different than the plants from which they originated, for example ground cumin and ground ginger.

In a further embodiment, the extraction process of the present invention can be used to extract a biologically active compound, such as a pesticide or a pharmaceutically active substance, or a precursor to such a compound from a raw material containing that compound or precursor, such as a plant material, a cell culture or a fermentation broth.

Accordingly, the present invention provides a process for extracting a composition comprising a biologically active compound or a precursor thereof from a raw material containing that composition as a constituent part, which process comprises the steps of (1) contacting a sample of the raw material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the raw material.

Suitable pesticides which may be extracted using the extraction process of the present invention include insecticides such as the pyrethroids.

Suitable pharmaceutically active substances which may be extracted using the extraction process of the present invention include the penicillins, the alkaloids, taxol, monensin and cytochalasin. Precursors to these compounds may also be extracted using the extraction process of the present invention. In one particular application for the extraction process of the present invention, taxol, which is an important anti-cancer drug, and/or taxane, which is a precursor to taxol, can be extracted from yew tree products, such as the bark or needles harvested from these trees.

According to a further aspect of the present invention, there is provided a composition comprising a pharmaceutically active substance obtained from a raw material product using the extraction process of the present invention.

According to a still further aspect of the present invention, there is provided a composition comprising a pharmaceutically active substance obtained from a raw material product using the extraction process of the present invention for use in medicine.

The present invention also provides a process for extracting a composition comprising one or more polar group containing compounds from a raw material containing that composition as a constituent part, such as a plant material, which process comprises the steps of (1) contacting a sample of the raw material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the raw material.

The extraction solvent which is used in the process of the present invention comprises a (hydro)fluorocarbon ether. Mixtures of two or more (hydro)fluorocarbon ethers may be used if desired. By the term (hydro)fluorocarbon ether we mean a compound selected from the group consisting of the hydrofluorocarbon ethers and the perfluorocarbon ethers.

(Hydro)fluorocarbon ethers having from 2 to 4 carbon atoms, especially the fluorine containing dialkyl ethers having this number of carbon atoms, are preferred, and of these the fluorine containing dimethyl ethers are particularly preferred. Examples of C_{2-4} (hydro)fluorocarbon ethers which may be useful in the extraction process of the present invention include, inter alia, bis(trifluoromethyl) ether (CF_3OCF_3), trifluoromethyl difluoromethyl ether (CF_3OCF_2H), pentafluoroethyl difluoromethyl ether ($CF_3CF_2OCF_2H$), 1,1,1,2-tetrafluoroethyl trifluoromethyl ether ($CF_3CFHOCF_3$), trifluoromethyl fluoromethyl ether (CF_3OCFH_2), bis(difluoromethyl) ether (CF_2HOCF_2H), pentafluoroethyl methyl ether

($CF_3CF_2OCH_3$) and 1,1,2,2-tetrafluoroethyl trifluoromethyl ether ($CF_2HCF_2OCF_3$).

The preferred (hydro)fluorocarbon ethers have a boiling point of 15°C or below, for example in the range of from -85 to 15°C, preferably 0°C or below, for example in the range of from -85 to 0°C, and more preferably -10°C or below, for example in the range of from -70 to -10°C.

In a preferred embodiment of the present invention, the extraction solvent which is used comprises a co-solvent in addition to the (hydro)fluorocarbon ether. Suitable co-solvents will typically have a boiling point of 60°C or below, for example in the range of from -85 to 60°C. The preferred co-solvents have a boiling point of 20°C or below, for example in the range of from -85 to 20°C, preferably 10°C or below, for example in the range of from -70 to 10°C, and more preferably 0°C or below, for example in the range of from -60 to 0°C. Mixtures of two or more co-solvents may be used if desired.

Suitable co-solvents may be selected from the C_{2-6}, particularly the C_{2-4} hydrocarbon compounds which may be aliphatic or alicyclic. Preferred hydrocarbons are the alkanes and cycloalkanes, with alkanes such as ethane, propane, n-butane and i-butane being especially preferred.

Other compounds which may be usefully employed as co-solvents in the extraction process of the present invention include the hydrocarbon ethers, particularly the dialkyl ethers, such as dimethyl ether, methyl ethyl ether and diethyl ether.

The extraction solvent preferably comprises from 50.0 to 99.5 % by weight, more preferably from 70.0 to 99.0 % by weight, and particularly preferably from 80.0 to 98.0 % by weight of the one or more (hydro)fluorocarbon ethers and from 50.0 to 0.5 % by weight, more preferably from 30.0 to 1.0 % by weight, and particularly preferably from 20.0 to 2.0 % by weight of the one or more co-solvents. If the co-solvent is a flammable material, which will be the case with the hydrocarbon and hydrocarbon ethers identified

above, then the extraction solvent will preferably comprise sufficient of the (hydro)fluorocarbon ether to render it non-flammable overall. Where the extraction solvent is a blend of one or more (hydro)fluorocarbon ethers and one or more co-solvents, the resulting blend may be zeotropic, but is preferably azeotropic or azeotrope-like. Azeotropic and azeotrope-like blends are preferred, since they behave essentially as a single substance.

The extraction solvent which is used in the process of the present invention may be in liquid, gaseous or vaporous form, but is preferably in liquid form.

The raw material which is subjected to the present extraction process may be a liquid, e.g. a solution, suspension or emulsion, or a solid. If the raw material is a solid, then the efficiency of the extraction process may be significantly improved by reducing the solid to a finely divided form, such as a powder.

The contacting of the extraction solvent with the raw material to be processed may be carried out under vigorous mixing conditions so as to facilitate the dissolution of the material to be extracted into the extraction solvent. The vigorous mixing may be achieved by mechanically shaking the extraction vessel containing the raw material/extraction solvent mixture or by stirring that mixture.

After the extraction process of the present invention has been completed, the solvent liquor containing the extract can be distilled to remove the extraction solvent from the extract. The resulting extract may then be used as it is or, alternatively, it may be subjected to one or more further processes, for example to purify the extract or to isolate a given compound or compounds contained in the extract.

In the preferred extraction process of the present invention, the extraction solvent which is used comprises a (hydro)fluorocarbon ether which has a relatively low boiling point compared to the extraction solvents used hitherto and, moreover, where a co-solvent is used this will likewise preferably have a relatively low boiling point. In consequence, once the extraction process of the present

invention has been completed to yield a solvent liquor containing the extract, the removal of the extraction solvent from the liquor tends to be relatively facile allowing the distillation to be carried out at relatively low temperatures, e.g. room temperature and below. This in turn reduces the risk of losing desired product either through co-evaporation of the more volatile compounds with the extraction solvent or thermal degradation of the more thermally unstable compounds.

The extraction process of the present invention may be operated continuously with the same extraction solvent being used repeatedly. A suitable installation for carrying out a continuous extraction process typically comprises an extraction vessel, a distillation unit, a compressor, a condenser and a suitable arrangement of connecting pipe work. The extraction solvent is first charged to the extraction vessel where it is contacted with the raw material to be processed, possibly under vigorous mixing conditions so as to facilitate the dissolution of the compound or composition to be extracted into the extraction solvent. The resulting solvent liquor containing the extract is then separated from the raw material, e.g. by allowing the liquor to drain through a filter arranged at the bottom of the extraction vessel, and passed to the distillation unit where the extraction solvent is removed by evaporation to leave the extract. The vapour generated in the distillation unit is compressed, e.g. using a diaphragm compressor, and is then delivered to a condenser which returns the extraction solvent to liquid form for recharging to the extraction vessel. With a continuous extraction process of this kind, it is possible to maximise the amount of the extract obtained without subjecting the same raw material sample to a succession of individual extractions. Once the raw material sample is exhausted, it is then removed from the extraction vessel and replaced with a fresh raw material sample.

Claims:

1. A process for extracting a compound or composition of matter from a raw material containing that compound or composition as a constituent part, which process comprises the steps of (1) contacting a sample of the raw material with an extraction solvent comprising a (hydro)fluorocarbon ether, and (2) separating the solvent liquor thus obtained containing the extract from the raw material.

Patents Act 1977 Examiner's report to the Comptroller under Section 17 (The Search report)	Application number GB 9503875.8
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Databases (see below) (i) UK Patent Office collections of GB, EP, WO and US patent specifications. (ii) ONLINE DATABASE: WPI	Documents considered relevant following a search in respect of Claims :- SINGLE CLAIM

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